Argonne National Laboratory

THE EBR-II MATERIALS-SURVEILLANCE PROGRAM:

II. Results of SURV-2

by

Sherman Greenberg, Robert V. Strain, and Earl Ebersole

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and

Earl Ebersole Idaho Facilities

Part I is ANL-7624

June 1970



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ABSTRACT

Eight subassemblies containing 15 alloys used in the primary system of EBR-II, and shield graphite canned in Type 304 stainless steel, are being irradiated in the EBR-II core at 700°F for varying periods of time and neutron dosage. Half of the alloy specimens are exposed directly to reactor sodium, and the remainder are sealed in helium. The alloys include AMPCO Gradel8 aluminum bronze; Stellite 6B; Inconel X-750; T-1 tool steel; Berylco-25; Types 304, 347, 416, and 420 stainless steel (the Type 304 in four variations); Type 17-4 PH stainless steel; and tantalum.

The second subassembly (SURV-2) has been removed and subjected to a limited examination after 1581 days in the reactor core and an estimated average fluence of 7×10^{19} n/cm² at energy levels >0.82 MeV (3×10^{21} n/cm² total). Of the materials exposed, only the Berylco-25 and tantalum experienced appreciable weight loss, and thus appear to be unsuitable for use in contact with sodium under the conditions of test. Type 304 stainless steel (from the shield-graphite cans) increased slightly in room-temperature yield strength but remained ductile. There was no evidence of loss of integrity of the canned-graphite neutron-shield structures. Tantalum samples experienced density decreases that were a function of fluence and equivalent to a maximum swelling of 0.4%.

I. INTRODUCTION

A long-term irradiation test program is in progress to monitor the behavior of the materials in service (primarily those in long-term service) in the primary-system sodium of EBR-II and of the neutron-shield graphite. The program involves the exposure and evaluation of 10 subassemblies containing the materials of interest in suitable form.

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A detailed description of the program and experimental method, and the results of SURV-1, have been reported.* The present report gives the results of a limited examination of SURV-2. This report is intended for use in conjunction with ANL-7624, which gives a detailed description of sample materials, types, and arrangements and experimental procedures. In particular, the loading diagram for SURV-2 is identical with that for SURV-1 (Table V, ANL-7624, p. 20).

Because the total fluence received by SURV-2 was below that thought to be necessary to significantly alter the mechanical properties of stainless steel, the mechanical-property determinations made for SURV-1 were not repeated for SURV-2. The graphite-can interaction has been studied in greater detail than for SURV-1, since the continued successful operation of EBR-II depends on the integrity of canned-graphite shield structures. The mechanical-property samples from SURV-2 have been stored so that measurements can be made in the future if desirable. In addition, high-temperature tensile properties of the Type 304 stainless steel tensile samples will be determined at Battelle Northwest Laboratories and reported separately.

II. DOSIMETRY AND EXPOSURE

SURV-2 was removed from the reactor on June 28, 1969. It had been in the reactor in position 12E1 for 1581 days at a temperature of approximately 700°F and had received a total exposure of 26,274 MWd. The estimated average total fluence, as calculated from one-dimensional, 22-group diffusion theory, was $3 \times 10^{21} \, \mathrm{n/cm^2}$.

Wire flux monitors, in reactor position 12E1, were used to determine the relative fast fluence as a function of position in the subassembly. The upper ends of the wires were 10.58 in. above the core centerline.

The neutron reaction for each of the four wires is:

 $\begin{array}{lll} \text{Copper:} & ^{63}\text{Cu}(n,\alpha)^{60}\text{Co} \\ \text{Iron:} & ^{54}\text{Fe}(n,p)^{54}\text{Mn} \\ \text{Nickel:} & ^{58}\text{Ni}(n,p)^{58}\text{Co} \\ \text{Titanium:} & ^{46}\text{Ti}(n,p)^{46}\text{Sc} \\ \end{array}$

Each wire was cut into 1-in. long sections. Each section was weighed, and then analyzed by gamma spectrometry (January 22, 1970). The activity in counts/min, summed under the photopeak and corrected for sample weight, was converted to dis/sec per gram of wire. The data are summarized in Figs. 1-4. The curves in the figures are useful in assessing the relative exposure of the samples to fast neutrons as a function of position in the subassembly.

^{*}The EBR-II Materials-surveillance Program: I. Program and Results of SURV-1, ANL-7624 (Sept 1969).

Each wire was cut into 1-in. long shotlows: Each section was woighed, and then analyzed by gament specification (issuery 22 1970). The activity in counts/min, successed under the plusteens and correctablest sample weight, who converted to dis/sec per grammal wire. The data are successed in Figs. 164. The corves in the injures are useful in sexpessing the relative expussion of the samples to last neutrons as a line to position of the subsembly.

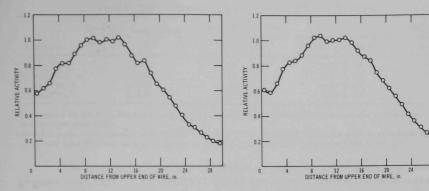


Fig. 1. ⁶⁰Co Activity of Copper Wire That Monitored SURV-2 Flux

Fig. 2. ⁵⁴Mn Activity of Iron Wire That Monitored SURV-2 Flux

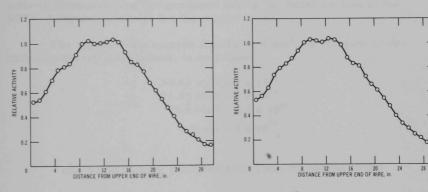


Fig. 3. ⁵⁸Co Activity of Nickel Wire That Monitored SURV-2 Flux

Fig. 4. ⁴⁶Sc Activity of Titanium Wire That Monitored SURV-2 Flux

The equilibrium activation rate at 45 MW has been calculated from the data for wire activity for a position ~1 in. above the core centerline, where the relative activity is taken as unity. The calculations are summarized in Table I. To correct for the intermittent operation of the reactor, the quantity

$$f\left(1 - e^{-\lambda t_1}\right) \left(e^{-\lambda t_2}\right)$$

was evaluated for each reactor run, where f = fraction of 45 MW exposure, t_1 = irradiation time, and t_2 = decay time from end of irradiation to counting time. The values of

$$f\left(1-e^{-\lambda t_1}\right)\left(e^{-\lambda t_2}\right)$$

the street and there and the days time in the contract of the

TABLE I. SURV-2 Neutron Activation Calculated from Flux Wires

Reaction	$\sum \left[f \left(1 - e^{-\lambda t_1} \right) \left(e^{-\lambda t_2} \right) \right]$	Activity, dis/sec per gram of Wire (1/22/70)	Equilibrium Activation Rate ^a at 45 MW ^b	Threshold Energy, MeV
54Fe(n,p)54Mn	0.2595	8.12 x 10 ⁶	5.38 x 10 ⁸	~ 3
58Ni(n,p)58Co	0.0767	3.97×10^{7}	7.61×10^{8}	~2.9
46Ti(n,p)46Sc	0.1008	8.22×10^{5}	1.03×10^{8}	-
63Cu(n,α)60Co	0.1475	2.67×10^{5}	2.62 x 10 ⁶	2 -

aCalculated by dividing the activity (column 3) by the summed term in column 2 and correcting for isotopic abundance.

bReactor power was increased to 50 MW in August 1968. However, since the bulk of exposure was at 45 MW (and SURV-1 data is in terms of 45 MW), these results are normalized to 45 MW.

The activation rate for the SURV-2 wires is approximately 30-40% greater than that for the SURV-1 wires because of the increase in core size (to accommodate an increased number of experiments) during the latter part of the irradiation period. Because the half-lives of the isotopes other than ⁶⁰Co are relatively short compared with the exposure time, the activity measured was that produced during the latter portion of the SURV-2 exposure (except for the copper wire).

The approximate neutron fluence in n/cm², as obtained from diffusion-theory calculations, is as follows:

$$>0.82 \text{ MeV}$$
--7 x 10¹⁹
>1.35 MeV--2 x 10¹⁹
>3.8 MeV--0.3 x 10¹⁹
Total--3 x 10²¹

III. RESULTS OF POSTIRRADIATION EXAMINATIONS

A. Weight-change Data

The weight-change data (obtained by using the hardness-sample cylinders) are summarized in Table II. As was true for SURV-1, only the Berylco-25 and tantalum samples showed consistently high weight losses. (There are no longer any Berylco-25 components operating in the reactor.) One sample each of the Ampco 18 aluminum bronze and T-1 tool steel showed unusually high weight losses. These weight losses are isolated cases, and components of these materials continue to perform satisfactorily in EBR-II. Therefore, it is believed that these unusual results (which will be subject to confirmation by examination of SURV-3) are due to experimental error and are not cause for concern.

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The activation rank for the SURV-1 wires because of the markage for the SURV-1 wires because of the markage macro core of the factor than that for accommodate an increased number of experiments) during the latter part of the irradiation period. Recease the half-lives of the isotopes of the relative period when the compared with the exposure time. The activity measured was that produced during the latter portion of the SURV-2 exposure (encept for the copper whre).

The approximate nestron fluence in norm, as obtained from diffusion-theory calculations, is as follows:

\$6.85 MeV - 2 x 10¹⁸ \$1.35 MeV - 2 x 10¹⁸ \$3.6 MeV - 0.3 x 10¹⁸

III. RESULTS OF POSTIRRADIATION EXAMINATIONS

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TABLE II. Weight Changes of Hardness Samples from SURV-2

Material	Sample ^a No.	Preirradiation Weight, g	Weight Change, b mg
Aluminum Bronze	A-1	16, 1235	-1.1
Ampco Grade 18	A-2	16.0560	-1.5
	A-3	16.0648	-9.7
	A-4	16.0629	-1.0
	A-5	16.0239	-0.0
Stellite 6B	B-1	18.0991	+0.3
neme ob	B-2		+0,3
	B-3	18.0889 18.0865	+0.1
	B-4		+0.1
	B-5	18.1008 18.1005	+0.6
		18.1005	
nconel X-750 (Heat-treated to hardness	C-1	17.7736	-1.1
f Rockwell C 37-40)	C-2	17.7620	-1.0
	C-3	17.7684	-1.1
	C-4	17.6870	-1.0
	C-5	17.7699	-1.2
ype 420 Stainless Steel (Heat-treated to	D-1	16.3578	-0.2
nardness of Rockwell C 40-45)	D-2	16.3498	-0.9
alditiess of Rockwell C 40-427	D-3	16.3557	-0.5
	D-4	16.4963	-0.9
	D-5		-0.5
		16.5042	
ool SteelT-1 (Heat-treated to hardness	E-1	18.6689	+0.2
f Rockwell C 55-60)	E-2	18.6770	-0.5
	E-3	18.6598	+0.1
	E-4	18.6753	-9.3
	/ E-5	18.7068	+1.1
247 Christers Charl	F-1	16.9494	-1.0
ype 347 Stainless Steel	F-2	16.9453	-0.4
			-0.4
	F-3 F-4	16.9346	-0.6
		16.9265	
	F-5	16.9272	-0.6
ype 416 Stainless Steel (Heat-treated to	G-1	16.3470	0.0
ardness of Rockwell C 30-34)	G-2	16.3083	+0.2
	G-3	16.3447	-0.3
	G-4	16.3548	-1.1
	G-5	16.3550	-0.7
deryllium CopperBerylco-25 (Heat-	H-1	17.9677	-137.7
	H-2	17.9745	-333.9
reated to hardness of Rockwell C 41-45)	H-3	17.9862	-491.6
	H-4	17.9594	-3.6
	H-5	17.9843	-1.1
ype 304 Stainless Steel with Boron	1-1	16.7669	-1.5
	1-2	16.7736	-1.1
	1-3	16.8037	-1.8
	1-4	16.5024	-0.7
	1-5	16.8292	-0.2
17 4 BU Strictor Street West treated	J-1	16.6606	-0.8
ype 17-4 PH Stainless Steel (Heat-treated	J-1 J-2		-0.8
hardness of Rockwell C 36-41)		16.6748	
	J-3	16.6930	-1.0
	J-4 J-5	16.6408 16.7058	-0.5 -0.6
ype 304 Stainless Steel	K-1	16.8490	-0.3
	K-2	16.8591	-1.8
	K-3	16.8633	-0.3
	K-4	16.8248	-0,3
	K-5	16.8222	-0.4
antalum	M-1	36.1254	-9.7
	M-2	36.1815	-29.9
	M-3	36.1617	-11.5
	M-4	36.1365	-18.4
	M-5	36.1470	+0.5

 3 For each material listed, the first four samples were exposed directly to the sodium of the reactor primary system. The fifth sample was sealed in a capsule containing helium. bExposed surface area was 7.8 cm 3 .

•		

B. Density-change Data

For all materials except Stellite 6B, Inconel X-750, T-1 tool steel, and tantalum, the postexposure densities were within the range measured before exposure.

Stellite 6B and Inconel X-750 increased in density by 13 and 17 mg/cm^3 , respectively. The initial densities were $8.376 \, ^{+0.003}_{-0.011}$ and $8.267 \, ^{+0.007}_{-0.003} \text{ g/cm}^3$, respectively.

The tool-steel samples decreased in density by from 14 to 18 mg/cm^3 . Initial density was $8.656 \pm 0.003 \text{ g/cm}^3$. Since there was no consistent relationship between density decrease and sample position (i.e., fluence), it is speculated that the change is the result of thermal conditions rather than neutron fluence.

The decrease in tantalum density $(54-77~\text{mg/cm}^3)$ was the greatest of any of the samples and varied directly with fluence. The maximum decrease in density was equivalent to a swelling of approximately 0.4%. The tantalum data (summarized in Table III) are of particular interest because of the relatively large change apparently caused by neutron exposure.

TABLE III. Density Decrease^a of Tantalum Hardness Samples from SURV-2

Sample No.	Environment	Position, in. above Bottom of Core	Density Decrease mg/cm ³
M-1	Sodium	1.00	61
M-5	Helium	1.00	54
M-2	Sodium	6.75	69
M-6	Helium	6.75	69
M-3	Sodium	15.50	63
M-7	Helium	15.50	55
M-4	Sodium	4.75	69
M-8	Helium	4.75	77

aThe average preexposure density was 16.685 $^{+0.005}_{-0.012}$ g/cm³.

There was essentially no difference between samples exposed to sodium and those exposed to helium.

C. <u>Hardness-change Data</u>

At the time of the preparation of the SURV-1 report (ANL-7624), hardness data for the samples exposed in helium were not available. The

C.Denstty-change Data

For all materials we call find the posterior with the range measured halofs extracted the range measured halofs extracted.

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The despess in tentulum dentity (54-77 mg/cm)) was the greatest of any of the despess in a consideration of the description and varied discrete with finences. The maximum decrease in dentity was equivalent to a condition of decrease the dentity was equivalent to a condition of the description of the condition o

TABLE III. De.sisp increases of Teninium Mardness Samples from SHRV-1.

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There was established the ended to be tween samples exposed to section and those exposed to be those

C. Hardness change Dall

At the time of the preparation of the SUR V-1 report (ANL-1649), hardness data for the mamples exposed its bellum were not available. The

Berylco-25 exposed to sodium had softened appreciably--a decrease in the average Vickers Hardness Number (500-g load) from 320 to the range of 161-176. In the absence of the helium-exposure data, it was speculated that the sodium environment contributed to the softening.

Since then, data from the helium-exposed samples from SURV-1 have become available. The average Vickers Hardness Number (500-g load) is 160. Thus, the sodium, although it severely corroded the Berylco-25, was not responsible for the softening. As stated in ANL-7624, the microstructure indicated that the Berylco-25 had overaged as the result of the SURV-1 exposure, and the overaging is responsible for the softening. A sodium-exposed Berylco-25 sample from SURV-2 had an average Vickers Hardness Number (500-g load) of 160; i.e., there is no significant change in hardness as a result of the increased exposure. It will be necessary to examine the samples exposed in the storage basket to distinguish between effects of thermal and neutron exposure.

D. Examination of Graphite and Cans

Measurement of external dimensions of the graphite cans showed no appreciable dimensional changes (to ± 0.001 in.) or torsional distortion (to ± 0.002 in.). When the cans were cut open, there was no evidence of sodium in-leakage to the graphite. In SURV-1, sodium leaked into the annulus between the cover plate and the end cap of one can. There was no such leakage of sodium in SURV-2.

1. Graphite

Table IV lists the density of the graphite after the SURV-2 exposure and the density of unirradiated material. The density of the unirradiated material was determined by weighing accurately measured machined blocks. This method was also used for SURV-1. However, it could not be used for SURV-2 because of mechanical difficulties, and the density was determined using a method based on ASTM Procedure C97-47 (Reapproved 1958) for natural building stone. Unirradiated canned graphite blocks have become available. Density of this graphite will be determined by using the ASTM procedure. Results will appear in the SURV-3 report.

TABLE IV. Effect of Exposure on Density of Graphite

Type of	Density,	g/cm^3
Graphite	Before Exposure	After Exposure
Plain	1.5419-1.6376 ^a	1.6933
Borated	1.5601	1.5639

aRange for six lots.

Borylos-25 exponent to medium had soffered appreciably -- a decrease in the average Vicesex Blandeses Number (200-g load) from 220 to the catige of 161-176, in the absence of the declars excessing data, it was speculated that the medium environment medium on the land of the softened to the softened to

Since their delicities the helium enquaed estables from SURV-1 have become evaluable, the average Micheses identically humber (800-g load) have become the vehicle, the average Micheses in AML first, the micro-was not responsible for the subsence the stated in AML first, the micro-structure indicated had the Derylovel had overaged as the estable of the SURV-1 exposites, and the overagings responsible to the sorieties. A subsence without the overage vehicle is a subsence with the derylovel and the sorieties when the sample vehicle is a subsence of the hardness humber for a lead in the first the subsence of the subsen

D. Examination of Graphite and Carls

Mensurement of external dimensions of the graphite cans showed no appreciable dimensional changes (to 40,002 in.) or inverting distortion (to ±0,002 in.). When the cans were on open, there was no evidence of wedlum in-leakage to the graphile. In SURV-1, sedium leakage to the graphile in SURV-1, sedium leakage of the can in SURV-2.

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exposite and the density of unifreedists a metafiel. The detailty of the uniformited interests and the detailty of the uniformited by weighing accurately encounted machined blocks. This method was also was a for SURV-1. However, if could not be used for SURV-2 because of receivables distributed the density was determined using a method based on ASTM Propedure C97-47 (Responded 1958) for material building store. Unitradiated extend graphite blocks have become sequence. Density of this graphite will be determined by main graphite will be determined by a single of the store that the store of the property of the store of the

TABLE IV. Eliect of Explante on Density of Gasphin

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Although the methods of measurement are different, it is likely that the increase in density is real and that there has been no swelling. An increase in density at exposures up to $\sim 1.5 \times 10^{22}$ nvt has been reported for somewhat comparable conditions in the Dounreay Fast Reactor.*

2. Can Material

Tensile samples (from the unwelded faces)** were cut and tested at room temperature. The results, together with values for similar samples cut from an unexposed can, are shown in Table V.

TABLE V. Tensile Properties of SURV-2 Graphite-can Material

		radiated Can Material	SURV-2 Irradiated Can Material	
Propertya	Average	Range	Average	Range
Yield strength, psi	39,400	38,300-40,400	42,500	40,900-44,100
Ultimate strength, psi	93,500	92,500-94,700	90,200	87,900-92,400
Total elongation, %	44	42-45	41	37-44
Reduction in area, %	57	56-59	50	45-56

aObtained at strain rate of 0.012 min⁻¹.

The SURV-2 exposure had only slight effect on roomtemperature tensile properties. The data for reduction in area and elongation show that the material is still very ductile after exposure in SURV-2.

Sections of the graphite cans, through the face welds, were bent 180° over a 1/2-in.-dia mandrel. The weld joint was parallel to the axis of the mandrel and at the apex of the bend. There was no cracking or other unusual effect.

The thickness of the carburized zone on the interior of the cans was similar to that after the SURV-1 exposure--~0.03-0.04 mm. However, in contrast with SURV-1, in which all graphite was easily removable from the cans, some sticking of the graphite occurred in SURV-2. Optical metallographic and electron-microprobe examination revealed no evidence of interaction between the graphite and stainless steel can material or of any unusual attack on the sodium-exposed surface of the stainless steel.

IV. DISCUSSION AND CONCLUSIONS

Examination of the materials exposed in SURV-2 indicate no materials-related problems or potential problems in the continued operation of EBR-II.

^{*}Reynolds, W. N., Physical Properties of Graphite, Elsevier Publ. Co., Amsterdam (1968), Ch. 7.

^{**}The can is formed by welding two half (channel-shaped) sections.

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TABLE W. Temple Proposite of SUNV-2 Graphic can Minight

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Type 304 stainless steel changed only slightly in room-temperature tensile properties and remained ductile after an exposure of 3 x $10^{21} \, \mathrm{n/cm^2}$ at $700^{\circ}\mathrm{F}$. Of more interest are the properties at reactor operating temperatures; these results will be determined at Battelle Northwest Laboratories, and the results will be made generally available.

As expected, Berylco-25 and tantalum continue to show poor corrosion resistance in sodium. However, all Berylco-25 components have long since been removed from the reactor, and the tantalum-clad neutron sources are being replaced. Corrosion of the tantalum source cladding has not produced a problem in the operation of the reactor.

Tantalum is the only material that has exhibited an appreciable reduction in density. No operational problems have resulted from this phenomenon. Samples from the sources may be useful in metallurgical studies.

The neutron-shield graphite structure continues to perform satisfactorily.

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